NIST Develops Algorithm for Robust Spectral Quantification of Large Particle Data Sets

NIST has developed a new algorithm that enables the research community to better take advantage of advances in scanning electron microscope-based automated particle analysis. With particle data sets now routinely exceeding ten-thousand particles, manual identification of the elemental constituents is not practical. NIST has led the effort in a new particle analysis methodology, and has developed the algorithms and tools necessary to produce the optimal analytical results from extremely large data sets.

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Until recently particle analysis with electron beam instruments, while capable of performing small-scale analyses, has been too time consuming to produce statistically significant population data. With the introduction of automated scanning electron microscopes optimized for rapid particle analysis it is now easy to collect ten-thousand particle or larger datasets. The first step in any quantification of a microanalytical spectrum is fitting the spectral peaks with measured or computed elemental reference spectra. Particle spectra are no different in this regard. However, from a practical perspective, the problem differs in both scale and character. When quantifying a small number of spectra it is practical to manually identify a minimal set of elements to fit. When you have hundreds or thousands of particles, this kind of manual effort is not practical. As a result, the analyst must select an exhaustive list of elements to fit against all spectra. The algorithm must then be smart enough to eliminate those elements that are not present based on the statistical evidence. In addition, particle spectra are different from classic bulk spectra. The count statistics in a particle spectrum fall between those of an x-ray map (very poor) and those of a classic microanalytical measurement (high quality.) Because each element can contribute more than one family of x-ray lines, the algorithm must be intelligent enough to consider all lines when making the determination. Yet because particle spectra can look very different from bulk spectra, many rules-of-thumb developed on bulk-spectra sometimes fail. With care, we can identify the presence or absence of elements in the few weight percent levels in spectra with twenty or thirty thousand counts. Finally, the algorithm must be robust enough to produce trustworthy results for thousands of spectra.

Samples run here at NIST and at other labs have identified a weakness in the current method of quantifying particle spectra. NIST is the primary laboratory leading this new particle analysis methodology, it is our role to develop the algorithms and tools necessary to produce the optimal results.

An analytical tool, named Graf, has been developed by NIST. It can reliably process data sets in excess of ten thousand particles against thirty or more elemental references.

In addition, Graf applies a tool (the max-pixel derived spectrum) developed for x-ray spectral imaging to the particle data set problem to facilitate the identification of the complete element set.

Future Plans: Many of the refinements developed in this project will be able to be applied back to classical electron probe analysis to address the long standing unresolved problem of performing reliable peak identification (qualitative analysis).

Figure 1: Graf spectrum plots showing the new algorithm's improvement over the old algorithm in its ability to discriminate the presence of minor elements. The new algorithm makes full use of the spectrum statistics to eliminate elements which lack sufficient evidence. The new algorithm was able to handle the difficult Pb-S overlap in this silver sulfide (Ag_2S) particle correctly.